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THE DETERMINATION OF COMBUSTIBLE MATTER IN SILICATE AND CARBONATE ROCKS

BY

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THE DETERMINATION OF COMBUSTIBLE MATTER IN SILICATE AND CARBONATE ROCKS.

By A. C. Fieldner, W. A. Selvig, and G. G. Taylor.

INTRODUCTION.

Chemists of the Bureau of Mines in the course of an investigation of methods of preventing explosions in coal mines by the use of rock dust had to determine accurately small percentages of combustible matter (organic carbon and organic hydrogen) in clay, shale, limestone, and other rocks for use in rock-dusting mines.[a] Owing to the presence of combined water in shale and clay, and of carbon dioxide in carbonate rocks, the percentage of combustible matter in these materials can not be calculated from the loss on ignition, as is done in coal and other fuels containing a relatively small proportion of constituents that are incombustible; neither can the combustible be taken as the sum of the total carbon and hydrogen as obtained in the ordinary ultimate analysis, for in both these methods the organic matter is oxidized to water and carbon dioxide, simultaneously with the liberation of water and carbon dioxide from the hydrous silicates and inorganic carbonates in the rock. Therefore, special methods are required that will differentiate between the organic and the inorganic carbon and hydrogen.

DETERMINATION OF ORGANIC CARBON AND ORGANIC HYDROGEN.

The percentage of organic carbon in carbonaceous rocks is easily obtained from a determination of the total carbon by the usual method of ultimate analysis[b] and a determination of the inorganic carbon by evolution with acid,[c] the difference between the values obtained in these two methods being the percentage of organic carbon present.

The determination of organic hydrogen involves a preliminary treatment of the sample whereby the hydrous silicates are decomposed, and their combined water is removed with the moisture, without effecting the organic compounds present.

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[a] Rice, G. S., and Jones, L. M., Methods of preventing and limiting explosions in coal mines: Tech. Paper 84, Bureau of Mines, 1915, pp. 15-21.

[b] Stanton, F. M., and Fieldner, A. C., Methods of analyzing coal and coke: Tech. Paper 8, Bureau of Mines, 1913, pp. 22-24.

[c] Parr, S. W., and Wheeler, W. F., Unit coal and the composition of coal ash: University of Illinois Eng. Exp. Sta. Bull. 37, 1919, pp. 28-29.

WORK OF LISSNER.

According to Lissner[a] this decomposition can be made by digesting the sample with a mixture of two parts by volume of fuming hydrofluroric acid (HF) and one part by volume of hydrochloric acid (HCl) with a specific gravity of 1.18, filtering off and washing the residue, drying it at 100° C., and determining the carbon and hydrogen in the usual way. Lissner first attempted to filter the residue through ignited asbestos, using suction, but found that the finely divided carbonaceous material quickly clogged the filter and made the filtration exceedingly slow. For this reason, in his later experiments, he used 9-cm. white ribbon No. 589, S. & S. papers, and corrected his results for the carbon and hydrogen from the filter paper.

In Table 1 are given the results obtained by Lissner with synthetic mixtures of coal and kaolin. In view of the correction for the carbon and hydrogen in the filter paper being larger than the amount of carbon and hydrogen in the sample of coal taken, the calculated and determined results agree unusually well. Seemingly the acid treatment removed all the hydrogen from the combined water in the kaolin without expelling any hydrogen or carbon from the coal substance. To avoid the necessity of correcting the results for carbon and hydrogen from the filter paper, Lissner finally adopted the procedure of carefully removing as much as possible of the washed carbonaceous residue from the filter paper, and merely determining the ratio of organic hydrogen to organic carbon. The organic hydrogen in the original substance was then computed from the total carbon determined in the untreated sample.

TABLE 1.—*Results obtained by Lissner in determining organic carbon and organic hydrogen in synthetic mixtures of kaolin and coal.*

Mixture No.	Description.	Carbon dioxide.				Water.			
		Total by analysis.	Less correction for filter paper.[b]	Calculated from analysis of constituents.	Loss by acid treatment.	Total by analysis.	Less correction for filter paper.[b]	Calculated from analysis of constituents.	Loss by acid treatment.
		Grams.	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*	*Grams.*
3	2.0744 g. kaolin 0.0210 g. bituminous coal	1.4248	0.6901	0.6905	0.0005	0.3145	0.0635	0.0633	−0.0002
4	1.2015 g. kaolin 0.2888 g. bituminous coal	1.6860	.9490	.9497	.0007	.3394	.0876	.0871	− .0005
5	1.0588 g. kaolin 0.2120 g. brown coal	1.4312	.6968	.6971	.0003	.3153	.0643	.0639	− .0004
6	1.0764 g. kaolin 0.2012 g. brown coal	1.3977	.6610	.6616	.0006	.3125	.0609	.0607	− .0002

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[a] Lissner, A., Zur elementar Analyse von tonhaltigen Kohlegesteinen; Chem. Ztg., vol. 34, 1910, pp. 37–38.

[b] The dry filter paper contained 6.18 per cent hydrogen and 44.41 per cent carbon.

Obviously, this calculated result will be in error if the material contains any carbonate. Hence it would seem essential always to determine the inorganic carbon by evolution with acid, and to deduct this value from the total carbon before multiplying by the ratio of organic hydrogen to organic carbon.

Moreover, there is a possibility of the heavier noncombustible material segregating on the filter and of bits of the filter paper being included in the sample, so that this method, in the opinion of the authors, is not nearly as satisfactory as a direct determination of the organic carbon and hydrogen after filtration through an incombustible filter such as asbestos, alundum, or platinum sponge. A series of experiments were therefore undertaken with incombustible filters to test the accuracy of the method with samples of coal, shale, kaolin and mixtures of known composition, the completeness of decomposition of hydrous silicates and the possible losses of organic carbon and hydrogen during the treatment with acids receiving special attention.

EXPERIMENTAL WORK.

REMOVAL OF INORGANIC HYDROGEN FROM KAOLIN AND SHALE.

A sample of washed North Carolina kaolin was air-dried and pulverized to a fineness of 100 mesh for use in testing the effectiveness of hydrofluoric acid treatment in removing combined water. One gram of this material was treated with 10 c. c. hydrochloric acid (1.18 sp. gr.) and 20 c. c. hydrofluoric acid (48 per cent HF) in a platinum dish on a water bath. After the dish had been heated 15 minutes, the kaolin was almost completely dissolved, leaving only a few gelatinous lumps that practically disappeared on heating the solution for 15 minutes more. Then the solution was evaporated to complete dryness, the dish remaining on the water bath an hour after becoming dry. This dry residue was treated with concentrated hydrochloric acid and digested until no further solution took place. After the liquid had been diluted with an equal volume of water and the digestion continued for some time, it was filtered with the aid of suction through ignited asbestos.

The filter and residue was washed free from chlorides, carefully transferred to a platinum boat and dried to constant weight in vacuum at 200° C., in the presence of phosphorous pentoxide (P_2O_5). Then the boat containing the dried residue was quickly transferred from the drying tube to the combustion furnace, and the carbon and hydrogen determined in the usual way. The following results were obtained:

	Carbon, per cent.	Hydrogen, per cent.
	0.20	0.35
	.25	.38
	.11	.26
Average.......	.19	.33

Obviously, the ratio of hydrogen to carbon is entirely too large for any possible organic compound; therefore, the greater part of the hydrogen must be inorganic hydrogen, probably a constituent of the insoluble precipitate that formed on evaporating the solution to dryness.

According to Abegg and Auerbach,[a] an aqueous hydrofluoric acid solution of alumina can not be evaporated to dryness without decomposition of the aluminum-fluorine compounds and the formation of difficultly soluble mixtures of alumina (Al_2O_3), aluminum fluoride (AlF_3), and double compounds of aluminum fluoride and hydrofluoric acid of the composition $Al_2F_6.HF.5H_2O$. If this compound was formed, it would be decomposed in the combustion furnace. The water of crystallization would all be absorbed in the calcium chloride tube and appear as hydrogen in the analysis. The hydrofluoric acid radical would also be split off by heat and appear partly in the calcium chloride tube and partly in the potash bulb.

In order to obtain some information on the actions of various acid concentrations on the formation of the insoluble aluminum fluoride compound, 1 gram of pure aluminum chloride was treated with a mixture of 10 c. c. of concentrated hydrochloric acid and 20 c. c. of strong hydrofluoric acid and evaporated to dryness on the water bath. Although this solution was clear at the beginning of evaporation, a gelatinous precipitate soon began to form and increased in quantity as the solution became more concentrated. The dry residue was difficultly soluble. Prolonged treatment with concentrated hydrochloric acid did not redissolve all of the residue.

Similar evaporations of aluminum chloride solutions with larger concentrations of hydrochloric acid and the same concentration of hydrofluoric acid seemed to retard the formation of insoluble matter. Therefore it seemed feasible to regulate the acid treatment and the relative concentrations of the two acids, so that complete decomposition of the hydrous silicates would take place and yet the formation of any appreciable quantity of insoluble hydrogen-bearing residue would be avoided. With this end in view, the North Carolina kaolin, and two shales of low combustible content were subjected to various methods of treatment by acids as described in Table 2. In each test the solution was filtered through ignited asbestos or platinum sponge. The filter and residue was dried at 105° C. and burned in the combination furnace in the usual manner.

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[a] Abegg, R., and Auerbach, Fr., Handbuch der anorganische Chemie, Bd. 3, Abteil. 1, 1905, pp. 71-72.

Table 2.—*Results obtained in determining organic carbon and hydrogen in kaolin and shales, showing the effect of various acid treatments on the decomposition of hydrous silicates.*

Description of acid treatment.	Kaolin, laboratory No. 22033.		Shale, laboratory No. 23089.		Shale, laboratory No. 23051.	
	Organic carbon.	Organic hydrogen.	Organic carbon.	Organic hydrogen.	Organic carbon.	Organic hydrogen.
(1)	(2)	(3)	(4)	(5)	(6)	(7)
	Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
Method A.—1 g. treated with 10 c. c. HCl[a] and 20 c. c. HF[a], in 100 c. c. platinum dish and evaporated to dryness on water bath. Residue then digested with 10 c. c. HCl, diluted with hot water and filtered through ignited asbestos. All operations conducted on water bath.	0.08 .07 .14 .23	0.52 .43 .64 .66	.... .46 .50	.... .25 .26	....	....
Method B.—1 g. digested 15 minutes on water bath with 10 c. c. HCl and 20 c. c. HF; 10 c. c. HCl then added, dish transferred to hot plate and rapidly evaporated to 5 c. c.; dish returned to water bath and evaporated to nearly dryness; 40 c. c. of (1:3) HCl then added, solution heated on hot plate 30 minutes, diluted with equal volume of hot water, boiled and filtered through ignited asbestos.	.01 .10 .06	.02 .00 .29	.50 .... .36	.05 .... .05	2.34 2.40 2.44	0.27 .25 .23
Method C.—1 g. digested 15 minutes on water bath with 10 c. c. HCl and 20 c. c. HF, and evaporated to 10 c. c. volume; 20 c. c. HCl then added, solution evaporated to 10 c. c. volume, diluted with 50 to 70 c. c. hot water, boiled and filtered through ignited asbestos. All operations on water bath.	.07 .04	.02 .01	.45 .61	.05 .07	2.35 2.38	.21 .22
Method D.—Same as method C, except that all heating was done on the hot plate instead of the water bath.	....	....	.39 .39	.11 .12	....	....
Method E.—Same as method D, except that the second evaporation with 20 c. c. HCl was omitted, i. e., no HCl other than the 10 c. c. in the original mixture being added.	....	....	.36 .25	.24 .15	....	....
Method F.—0.5 g. sample digested 5 minutes on hot plate with 15 c. c. HCl; 20 c. c. HF then added and solution digested just below boiling point for 2 hours, adding more HCl and HF, to maintain volume above 15 c. c.; finally, solution was evaporated to 15 c. c. volume, 25 c. c. HCl added, boiled and filtered through asbestos or platinum sponge.	....	....	.33 .46 .50	.18 .07 .07	.... 2.43	.... .30

[a] Concentrated HCl of 1.19 specific gravity and 48 per cent HF were used when strength of acid is not shown.

DISCUSSION OF RESULTS.

The results of these experiments, as given in Table 2, show that it is possible to conduct the acid treatment so that no appreciable amount of inorganic hydrogen or inorganic carbon is retained in the insoluble residue. The kaolin that contained no organic matter went into complete solution on digesting with a mixture of 10 c. c. of concentrated hydrochloric acid and 20 c. c. of strong hydrofluoric acid. On concentrating this solution, either on the hot plate or on the water bath, a white crystalline precipitate formed; this precipitate redissolved on adding concentrated hydrochloric acid before the solution had gone to complete dryness. With this procedure, in methods B and C (Table 3), no appreciable amounts of carbon or hydrogen were obtained in the slight residue that was filtered from the diluted solution.

Evaporation to complete dryness yielded an insoluble residue that gave, on analysis, 0.4 to 0.7 per cent hydrogen. Similar results were obtained in the treatment of the two samples of shale that contained small amounts of combustible matter. Evaporation to complete dryness resulted in the formation of an insoluble residue containing some inorganic hydrogen, as shown in the high hydrogen values obtained by method A. It was possible to redissolve all but traces of the insoluble aluminum fluoride compound by adding an excess of hydrochloric acid to the solution after the silicate was decomposed, and before complete dryness was reached; and, as equally good results were obtained with methods B, C, and F, it was evident that treatment on the water bath was just as effective in decomposing the silicate as the higher temperature attained in digesting on the hot-plate. Obviously, the carbonaceous rocks should be decomposed at the lowest possible temperature to minimize loss of organic matter.

LOSS OF ORGANIC CARBON AND HYDROGEN DURING ACID TREATMENT.

In order to determine what action the various acid treatments has on the organic matter in coal or carbonaceous shale, a sample of clean bituminous coal containing 1.24 per cent ash was selected for treatment by the same methods as were found effective in decomposing kaolin and shale. Because of the low ash content, any possible inorganic hydrogen from hydrous silicates in this coal must necessarily be less than 0.01 per cent; therefore the total percentage of hydrogen less the hydrogen in the moisture of the coal should be the same as that obtained by combustion of the treated and dried residue if there is no loss through the acid treatment. The results of these experiments are given in Table 3.

TABLE 3.—*Effect of various acid treatments on the coal substance of semibituminous coal (Lab. No. 22933).*

[Analysis: Inorganic CO_2 by evolution with acid=0.03; moisture=2.53; ash=1.24; total carbon=84.76; total hydrogen=4.11; probable water of composition in hydrous silicates of inorganic matter=0.05×1.24=0.06;
total inorganic hydrogen in coal =1/9 (2.53+0.06)=0.29;
theoretical organic hydrogen =4.11—0.29=3.82;
theoretical organic carbon =84.76—0.01=84.75.]

Amount of sample taken. (1)	Acid treatment. (2)	Determined by combustion after acid treatment.		Loss due to acid treatment.	
		Organic carbon. (3)	Organic hydrogen. (4)	Organic carbon. (5)	Organic hydrogen. (6)
Grams.		*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
0.2000	Method B[a]	84.15	3.63	0.60	0.19
.2000	.....do	83.67	3.69	1.08	.13
.2000	.....do	84.22	3.66	.53	.16
.2000	.....do	83.74	3.62	1.01	.20
.2000	Method C	83.92	3.63	.83	.19
.2000	.....do	84.13	3.69	.62	.13
.2000	Method D	82.05	3.45	2.70	.37
.2000	.....do	82.17	3.41	2.58	.41
.2000	Method E	84.01	3.62	.74	.20
.2000	.....do	84.01	3.60	.74	.22
.2000	Method F	84.28	3.66	.47	.16
.2000	.....do	84.23	3.65	.52	.17

[a] See Table 2 for description of methods.

In each experiment there was a small but measurable loss of hydrogen and carbon. The lowest loss of 0.50 per cent carbon and 0.17 per cent hydrogen was found in the samples treated by method F, which involved digesting the sample on the hot plate for a period of two hours at a temperature just below the boiling point and maintaining the volume of the liquid above 15 c. c.

Two evaporations on the water-bath to a volume of 10 c. c., according to method C, also gave small losses, averaging 0.68 per cent carbon and 0.15 per cent hydrogen, a single evaporation to 10 c. c. on the hot-plate, by method E, gave slightly greater losses, averaging 0.74 per cent carbon and 0.21 per cent hydrogen; and two evaporations to 10 c. c. on the hot-plate, according to method D, increased the losses decidedly. The average losses by method D were 2.64 per cent carbon, and 0.39 per cent hydrogen. As compared with methods C and F, method B, in which the evaporation was carried to small volume on the hot-plate and finished on the water-bath, showed small losses in two determinations and larger losses in two other determinations, possibly due to higher hot-plate temperatures.

Hence one may conclude from these experiments that the minimum loss of organic hydrogen and carbon is associated with the shortest time of digestion and lowest temperature of treatment that will serve to decompose the hydrous silicates in the mixture. Evaporation to concentrated solution on the hot-plate is to be avoided, as it subjects the carbonaceous material to higher temperatures as the concentration and boiling point of the solution increases. These precautions apply more especially to materials containing a large proportion of combustible matter.

PROCEDURE FOR DETERMINING ORGANIC CARBON AND HYDROGEN IN SILICATE ROCKS.

A sample weighing 0.2000 to 1 gram of the finely ground rock, the weight taken depending on the relative proportion of combustible, is placed in a 100-c. c. platinum dish with 15 c. c. of concentrated hydrochloric acid and heated on the hot-plate 10 minutes; then 20 c. c. of strong hydrofluoric acid is added and the dish is heated at a temperature of incipient boiling until the inorganic matter seems to be decomposed. From one-half hour to two hours will be required for decomposition. More hydrochloric and hydrofluoric acids, in these proportions, should be added from time to time to maintain the volume of the liquid above 15 c. c.

When the decomposition appears complete, the liquid is evaporated to a volume of 15 c. c.; then 25 c. c. of concentrated hydrochloric acid is added and the solution is heated to boiling to redissolve the acid aluminum fluoride. An equal volume of hot water is added to the boiling solution and the liquid is again heated to boiling, after which it is filtered, while hot, through a filter of ignited asbestos or platinum sponge, and is washed free from chlorides with hot water. The filter and residue are transferred to a porcelain or platinum boat and dried two hours at 105° C.; then the boat containing the residue is transferred directly from the drying oven to the tube of the combustion furnace so that no moisture may be reabsorbed. The percentages of carbon and hydrogen are determined in the usual manner. In heating the solution on the hot-plate, care must be taken not to carry the evaporation to a point where the volume is less than 15 c. c. or the temperature is higher than that of incipient boiling. If the sample is chiefly of organic matter, conducting the digestion on a water-bath is preferable.

PREPARATION OF ASBESTOS FILTER.

In some of the experiments described in this paper the authors used platinum sponge[a] for the filtering medium. A mat of platinum sponge was formed in a platinum boat having a perforated bottom, and the solution was filtered through this boat with the aid of suction. However, this filter did not prove as convenient as a simple carbon filter of ignited asbestos, prepared in the following manner:

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[a] Monroe, C. E., Filtration with filters of metallic felt: Jour. Anal. Chem., vol. 2, 1888, p. 241.

Figure 1 shows the form of filtering apparatus described by Lord and Demorest.[a] *a* is a 500-c. c. heavy glass filtering flask which is connected by the side tube to a suction pump. *b* is a glass tube having a uniform bore of about $\frac{5}{8}$ inch and about 9 inches long, which passes through the rubber stopper in the neck of the flask. Into this tube a second tube *c* is inserted, just large enough to slip freely through the larger tube; it should be 1 inch longer than the larger tube and should have the ends ground off square. The apparatus should be so arranged that when the smaller tube rests on the bottom of the flask about two-thirds of the upper tube will be unoccupied. *d* is a perforated disk of sheet platinum, small enough to slip freely into the larger tube and rest in place on the end of the inner tube. *e* is a piece of stiff platinum wire, gold soldered to the disk. Instead of the wire a piece of glass rod may be fastened in the center of the disk to serve as a handle.

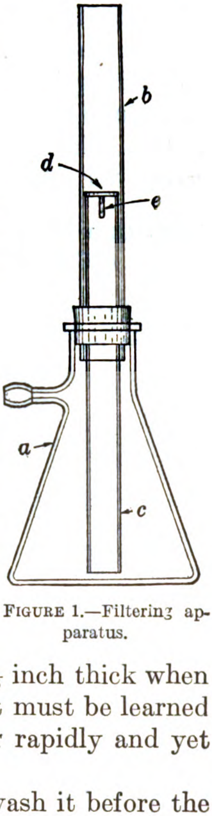

FIGURE 1.—Filtering apparatus.

The ignited asbestos for the felt is prepared as follows: Long-fibered chrysolite asbestos is cut across the fibers into pieces $\frac{3}{16}$ inch long. These pieces are ignited in a crucible or dish at a low red heat for at least 30 minutes. When the asbestos is cool, it is transferred to a clean porcelain mortar and macerated to a pulp with strong hydrochloric acid. This paste is diluted with a large amount of water, poured into a tall beaker, and allowed to settle until the fibrous mass collects at the bottom, leaving the fine milky silt in suspension. All this fine milky material is removed by repeated washing and decantation until the wash water becomes practically clear. Asbestos prepared in this manner makes a felt that filters rapidly. It is preserved in water in a stoppered bottle.

The filter is formed by pouring enough of the suspended asbestos into the tube to form a layer $\frac{1}{16}$ inch thick when drawn down by suction. Exactly the right amount must be learned by experience. A properly prepared felt will filter rapidly and yet retain the finest precipitate.

A little water is first drawn through the felt to wash it before the solution is poured on. After the filtration and washing are completed, the stopper, with the tubes, is taken out of the flask, the smaller tube is washed off, and then pushed carefully through the larger tube in order to force out from below the disk carrying the felt and residue. By using care no residue will be left adhering to the tube. The filter pad and residue are easily detached from the platinum disk. They are put in a porcelain or platinum boat for drying and subsequent combustion. Should any residue adhere, it must be wiped off with a tuft of ignited asbestos and transferred to the boat containing the felt from the platinum disk.

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[a] Lord, N. W., and Demorest, D. J., Metallurgical analysis: 3rd ed., New York, 1913, pp. 118–120.

PROCEDURE FOR DETERMINING ORGANIC CARBON AND HYDROGEN IN CARBONATE ROCKS.

Because of the formation of comparatively insoluble fluorides, such as $CaF_2.2HF.6H_2O$, containing water of crystallization, the treatment of carbonate rocks must be so modified as to remove the alkaline earth elements that are combined with the carbonate radical. This removal is readily accomplished by treating the sample with 30 c.c. of dilute (1:2) hydrochloric acid and heating it until all the carbonates are decomposed. Then the solution is diluted with water, filtered through ignited asbestos in the carbon filter tube, and washed with acidulated water. The filter and the residue are transferred to the 100-c.c. platinum dish and treated exactly as described in the procedure for silicate rocks.

RESULTS WITH SYNTHETIC MIXTURES OF COAL AND KAOLIN, AND OF COAL, KAOLIN, AND CALCIUM CARBONATE.

In Table 4 are given some results obtained in determining the organic carbon and the organic hydrogen in synthetic mixtures of coal and kaolin, and of coal, kaolin, and calcium carbonate.

Coal No. 23149, a semibituminous coal from the New River field, West Virginia, contained by ultimate analysis 0.63 per cent moisture, 7.70 per cent ash, 82.09 per cent carbon, and 4.44 per cent hydrogen (after deduction of 0.07 per cent hydrogen present as moisture). Assuming that the combined water content was equivalent to 5 per cent of the ash, another deduction of 0.04 per cent may be made from the hydrogen, giving a net organic hydrogen content of 4.40 per cent in the coal as used in the synthetic mixtures. As the coal contained no carbonates, the total carbon content of 82.09 per cent may be considered organic.

Coal No. 22933, a selected sample of semibituminous coal from the Bering River field, Alaska, analyzed as follows: Moisture 2.53 per cent, ash 1.24 per cent, carbon 84.76 per cent, hydrogen 3.83 per cent (after deduction of 0.28 per cent hydrogen present in the moisture). As the coal contained no carbonates and only 1.24 per cent ash, all of the carbon and hydrogen must be organic.

Kaolin No. 22033, a washed sample from North Carolina, contained no combustible matter, and 14.54 per cent of moisture and combined water. The calcium carbonate contained no organic matter.

TABLE 4.—*Results obtained in determining the percentages of organic carbon and organic hydrogen in synthetic mixtures of coal and kaolin, and coal, kaolin, and calcium carbonate.*

Mixture No.	Description.	Organic carbon.			Organic hydrogen.		
		Calculated from analysis of constituents.	Determined.	Difference.	Calculated from analysis of constituents.	Determined.	Difference.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
1	0.2 g. coal, No. 22933	24.21	24.11	−0.10	....	....	....
	0.5 g. kaolin, No. 22033	....	....	....	1.09	1.06	−0.03
1	0.2 g. coal, No. 22933 0.5 g. kaolin, No. 22033	24.21	24.05	− .16	1.09	1.06	− .03
1	0.2 g. coal, No. 22933 0.5 g. kaolin, No. 22033	24.21	24.08	− .18	1.09	1.06	− .03
2	0.95 g. kaolin, No. 22033 0.05 g. coal, No. 23149	4.10	4.05	− .05	.22	.25	+ .03
2	0.95 g. kaolin, No. 22033 0.05 g. coal, No. 23149	4.10	4.22	+ .12	.22	.27	+ .05
3	0.85 g. $CaC0_3$ 0.10 g. kaolin, No. 22033 0.05 g. coal, No. 23149	4.10	3.97	− .13	.22	.22	....
3	0.85 g. $CaC0_3$ 0.10 g. kaolin, No. 22033 0.05 g. coal, No. 23149	4.10	4.09	− .01	.22	.23	+ .01

It is apparent from the excellent agreement between the calculated and determined results (Table 4) obtained with all the synthetic mixtures, that all the inorganic carbon and hydrogen from the kaolin and calcium carbonate was effectively removed without any material loss of hydrogen or organic carbon being sustained during the acid treatment.

RESULTS OF ANALYSES OF VARIOUS SHALES, CLAYS, AND LIMESTONES.

In Table 5 (p. 17) are given the results obtained in the determination of total and organic carbon and hydrogen in various shales, clays, and limestones. These analyses illustrate the wide application of the methods just described in determining the true combustible content of materials that contain carbon and hydrogen in both combustible and incombustible form. It should be noted that the difference between the percentages of total and organic carbon agrees very well with the percentage of carbonate carbon as determined by evolution with acid. This agreement is another check on the accuracy of the method.

METHOD OF COMPUTING AND REPORTING ROCK ANALYSES.

The following analyses of a roof shale illustrates the determinations that are required in the chemical examination of a rock with reference to its suitability for rock-dusting.

Analysis of air-dry sample of shale.

	Per cent.		Per cent.
Moisture	0. 35	Total carbon	3. 67
Ash	91. 85	Organic carbon	3. 24
Sulphur	. 32	Total hydrogen	. 58
		Organic hydrogen	. 25

From these determinations the carbon dioxide present in carbonates and the combined water of the hydrous silicates are calculated as follows:

$CO_2 = \frac{11}{3}$ (total C − organic C) $= \frac{11}{3}$ $(3.67 - 3.24) = 1.58$ per cent.

Combined water $= 9$ (total H − organic H) − moisture $=$
$9\ (0.58 - 0.25) - 0.35 = 2.62$ per cent.

With the aid of these computed values the complete rock analysis is reported as follows:

Composition of air-dry shale.

[Sample of roof shale. Lab. No. 24225.]

	Per cent.	
Incombustible:		
Moisture	0. 35	
Ash	91. 85	
Carbon dioxide	1. 58	
Combined water	2. 62	
Total (per cent)		96. 40
Combustible:		
Organic carbon	3. 24	
Organic hydrogen	. 25	
Sulphur	. 32	
Total (per cent)		3. 81
		100. 21

When a number of samples of rock from the same source are to be analyzed for combustible content, the total and organic carbon and hydrogen determinations need be made on one sample only, from which the ratio of organic carbon to organic hydrogen may be calculated. As this ratio will be fairly constant for rocks from the same source, it is merely necessary to determine the moisture, ash, sulphur, carbon dioxide by evolution with acid, and the total carbon and hydrogen by the usual method of ultimate analysis. The organic carbon will then be the difference between the total carbon and the carbonate carbon ($\frac{3}{11}$ of CO_2), and the organic hydrogen will be the organic carbon multiplied by the ratio of organic hydrogen to organic carbon.

COMBINED WATER IN SHALES AND CLAYS FROM COAL MINES.

In Table 5 are given the percentages of combined water in a number of shales, clays, and other rocks from coal mines. These values are of interest in connection with the factor for hydration proposed by Parr,[a] in his formula "Total inorganic matter = moisture + 1.08 ash + 22/40 sulphur," for calculating the total inorganic matter in coal. The factor of 1.08 used in this formula appears somewhat high in comparison with the values obtained for combined water in the shales and clays listed in Table 5. In none of these was the percentage of combined water more than 6 per cent. The actual values obtained in the various shales and clays from different mines ranged from 2.5 to 5.7 per cent. The average was about 5 per cent.

TABLE 5.—*Organic carbon and hydrogen found in shales, clays, and limestones.*

[**Analyses computed to moisture-free basis**].

Laboratory No. (1)	Description. (2)	Carbonate carbon determined by evolution with acid. (3)	Total carbon (4)	Organic carbon. (4)	Difference. (6)	Total hydrogen. (7)	Organic hydrogen. (8)	Difference. (9)	Water of composition. (10)	Ratio of organic carbon to organic hydrogen. (11)
		Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
23150	Pit shale No. 1 from experimental mine, Allegheny County, Pa.	0.25	0.52	0.26	0.26	0.07	0.04	0.03	5.67	6.5
23080	Pit shale No. 1 from experimental mine, Allegheny County, Pa.	.23	.76	.45	.31	.64	.05	.59	5.22	9.0
23051	Pit shale No. 1a from experimental mine, Allegheny County, Pa.	.21	2.78	2.44	.34	.76	.25	.51	4.59	9.8
23997	Shale from Vesta No. 4 mine, Washington County, Pa.	.06	11.94	11.50	.44	1.27	.67	.60	5.40	17.2
24303	Shale partings from Margaret No. 2 mine; Harkness bed, St. Clair County, Ala.	......	......	5.68	......	......	.89	......	......	6.4
23577	Rock partings from Margaret No. 2 mine; Harkness bed, St. Clair County, Ala.	......	3.93	3.39	.54	.83	.35	.48	4.32	9.7
24225	Roof shale No. 4 from Girard mine No. 7, Crawford County, Kans.	.46	3.68	3.25	.43	.54	.26	.28	2.52	12.5
24085	Roof shale, black "slate," from Vandalia mine No. 82, bed No. 5, Vigo County, Ind.	1.81	18.93	17.25	1.68	1.65	1.36	.29	2.61	12.7
24084	Bottom clay, black "slate" from Vandalia mine No. 82, bed No. 5, Vigo County, Ind.	.05	2.13	1.96	.17	.68	.13	.55	4.95	15.1
24454	Floor material from Gebo mine, Hot Springs County, Wyo.	.01	4.66	4.62	.04	.67	.35	.32	2.88	13.2
22033	Washed kaolin from North Carolina.	.01	.08	.07	.01	1.53	.01	1.52	13.68	7.0
24608	Limestone dust used in experimental mine, Pittsburgh experiment station	10.35	......	.13	......	......	.06	......	......	2.2
23886	Marble dust from Tennessee.	11.92	......	.33	......	......	.03	......	......	11.0

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[a] Parr, S. W., and Wheeler, W. F., Unit coal and the composition of coal ash: Bull. 37, University of Illinois Eng. Exp. Sta., 1909, p. 35.

SUMMARY.

1. Lissner's method of determining organic carbon and hydrogen gave abnormally high results for organic hydrogen in rocks containing small amounts of combustible matter. On digesting a sample of pure kaolin containing no combustible matter with a mixture of hydrochloric and hydrofluoric acids, and evaporating the solution to dryness, an insoluble crystalline precipitate containing aluminum fluoride, hydrofluoric acid, and probably water of crystallization was formed.

2. The formation of this residue can be prevented by adding an excess of hydrochloric acid after the silicates are decomposed by the hydrofluoric acid mixture, and by not carrying the evaporation to dryness.

3. Experiments with bituminous coals of low ash content showed that a small but measurable loss of organic carbon and hydrogen occurred on digesting pure coal with a mixture of hydrofluoric acid and hydrochloric acid. This loss could be kept down to negligible proportions by conducting the digestions at temperatures not above that of incipient boiling, or by using the water bath only.

4. Experiments with calcareous rocks showed that the carbonate must be decomposed and the calcium removed before digesting the material with hydrofluoric acid, otherwise insoluble calcium fluoride and acid calcium fluoride ($CaF_2 . 2HF. 6H_2O$) are formed.

5. Analyses of shales, clays and slates from coal mines showed ratios of organic carbon to organic hydrogen, ranging from 7 to 17. The combined water [combined water $=9$ (total H $-$ organic H) $-$ moisture] varied from 2.5 to 5.7 per cent.

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